MISSOURI DEPARTMENT OF TRANSPORTATION
MATERIALS ENGINEERING
Jefferson City, Missouri

Test Method
MoDOT T25
DETERMINATION OF CHLORINE IN CHLORINATED RUBBER AND CHLORINATED PARAFFIN

1.0 SCOPE

This method describes a procedure for determining the percent Chlorine in Chlorinated Rubber and Chlorinated Paraffin used in traffic paint vehicle.

2.0 REAGENTS AND APPARATUS

(a) Parr Peroxide Bomb Apparatus, 22 mL, flame ignition Macro Bomb, including ignition shield.

(b) Sodium Peroxide (Na_2O_2), Reagent Grade, granular, low chloride.

(c) Nitric Acid (HNO_3), Reagent Grade.

(d) Silver Nitrate ($AgNO_3$), Reagent Grade.

(e) Sodium Chloride (NaCl), Reagent Grade, dried at 105-110 C for 1 hour prior to use.

(f) Millivolt Meter equipped with a Combination Chloride Electrode.

3.0 PREPARATION OF STANDARD SOLUTIONS

(a) Standard Sodium Chloride Solution (0.0100N) - Weigh 0.5844 gm dried NaCl, Reagent Grade, dissolve in water and dilute to 1L.

(b) Standard Silver Nitrate Solution (0.01N) - Weigh 1.699 gm $AgNO_3$, Reagent Grade, dissolve in water and dilute to 1L. Standardize against 0.0100 N NaCl.

4.0 PROCEDURE

(a) Weigh, to the nearest 0.1 mg., 0.40 gm sample for Chlorinated Paraffin or 0.30 gm sample for Chlorinated Rubber and transfer to a Parr Bomb cup which contains 15 gm Na_2O_2. Thoroughly mix the contents of the fusion cup with a stirring rod, wipe the rod with a small piece of filter paper and place the paper in the cup. Place the bomb assembly in the protective shield and fire strongly with a Bunsen burner for 20 minutes. Cool to room temperature and disassemble. (NOTE: If difficulties arise in obtaining complete combustion of Chlorinated Paraffin samples, small amounts of powdered sucrose or powdered $NaNO_3$ may be mixed with the Na_2O_2 and sample the fusion cup prior to ignition. However, the total combustible matter should not exceed 0.5 gm.) Rinse the bomb cover into a 600-mL beaker with about 150 mL of water, place the cup on its side in the beaker, cover with a watch glass and warm gently until solution of the fused material is complete. Remove the cup and rinse well with water. Add HNO_3 in small increments until the solution is acid to methyl orange. Add 5 mL HNO_3 in excess and warm gently until solution is complete. Filter through a coarse paper into a 1000-mL volumetric flask, washing well with hot water. Allow to cool to room temperature and dilute to volume with water.

(b) Transfer a 50-mL aliquot to a 250-mL beaker and determine percent chlorine by potentiometric titration.

(c) Perform a blank determination following the procedure given in (a) and (b) above, omitting only the addition of sample.

5.0 CALCULATION AND REPORT

Report percent chlorine to the nearest 0.1% as follows:

$$\% \text{ Chlorine (Cl)} = \frac{(A - B) \times C \times 0.709}{D} \times 100$$

Where:

A = milliliters of $AgNO_3$ solution to titrate sample,
B = milliliters of $AgNO_3$ solution to titrate blank,
C = normality of $AgNO_3$ solution,
D = grams of the sample.